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Toughening of a Particulate-Reinforced/Ceramic-Matrix Composite

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# **Abstract**

The micromechanics involved in increased crack growth resistance,  $K_R$ , of a TiB2 particulate-reinforced/SiC-matrix composite over that of a monolithic SiC was analyzed experimentally and theoretically. Fractography studies showed that the advancing crack was attracted to the particulates due to the tensile region surrounding the particulate. Countering this effect was the compressive thermal residual stress in the matrix, which resulted in the toughening of the composite. This thermal residual stress field in a particulate reinforced ceramic matrix composite is induced by the mismatch in the coefficients of thermal expansions of the matrix and the particulate when the composite was cooled from the processing to room temperatures. The increase in,  $K_R$ , of the TiB2-particulate/SiC-matrix over the monolithic SiC matrix, measured by using an hybrid experimental-numerical analysis, was 77 percent, and compared well with the analytically predicted increase of 52 percent. The increase in  $K_R$  predicted by the crack deflection model was 14 percent. Dependance of  $K_R$  on the volume fraction of the particulates and of voids is also discussed

# I. <u>Introduction</u>



The primary toughening mechanisms of particulate reinforced ceramics have been attributed to: (i) interaction between the crack front and particulates (crack front bowing model)<sup>1-2</sup>; (ii) crack deflection by the particulates in front of a propagating crack (crack deflection model);<sup>3</sup> and (iii) crack bridging by ductile particulates (particulate bridging model).<sup>4</sup> Other secondary mechanisms which contribute to the toughening of ceramic composites are: (iv) residual stress (strain) field due to mismatch between the coefficients of thermal expansion (CTEs) of the ceramic matrix and particulates;<sup>5,6</sup> and (v) the grain size.<sup>7</sup>

Increase in the fracture toughnesses,  $K_c$ , of particulate-reinforced/ceramic-matrix composites with modest particulate volume fractions,  $f_p \equiv 0.1 \sim 0.3$ , over the fracture toughness of unreinforced ceramic matrix,  $K_m$ , vary from 10 percent<sup>7</sup> and 40 to 70 percent<sup>5,6,8,9</sup>. This increase in the fracture toughness was attributed to crack front deflection. Several investigators have observed such crack deflection due to particulates in particulate reinforced ceramic matrix composites.<sup>3,5,8</sup> Such crack deflection was also observed in monolithic ceramics and was considered a major contributor to the toughening of ceramics, <sup>10</sup> although the crack deflection in monolithic ceramics takes place along the grain boundary. The crack deflection observed in particulate-reinforced/ceramic-matrix composites, however, was much larger in magnitude, and was thus considered a dominant toughening mechanism.

The crack deflection model of Faber and Evans<sup>3</sup> is based on a geometrical treatment of a crack deflected from its main crack plane and ignores the local stress field at and near the interface between the matrix and the filler (particulate in this paper). This local stress is caused by the difference in the stiffnesses and/or the coefficient of thermal





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expansion (CTE) between the matrix and particulate. The crack deflection model predicts the toughness ratios of  $K_c/K_m = 1.12\sim1.15$  for uniformly distributed particles and for particle volume fractions of  $f_D = 0.1 \sim 0.3$ . This  $K_c/K_m$  ratio predicted by the crack deflection model is smaller than 1.4-1.7, typically observed in most particulate reinforced ceramic matrix composites. 5,6,8,9 Thus, the crack deflection model only accounts for a portion of the increase in the toughness. An additional toughening mechanism which has been identified in fiber-reinforced/ceramic-matrix composite is fiber bridging in the region behind a crack tip. 11-15 The fiber bridging model, however, is only applicable to short and continuous fiber composites and not to a particulate reinforced composite. A similar idea has recently been used by Budiansky et al. (1988) to account for the toughening of a ceramic by the bridging of ductile metal particulates, which deform elastically/plastically until failure. This model is valid for ductile metal particulates, but is not valid for brittle particulates such as an intermetallic compound, for example, TiB2. Moreover, it is much less likely that TiB2 particulates located on the crack plane will break.

An additional mechanism, which can contribute to the toughening of a particulate-reinforced/ceramic-matrix composite, is the thermal residual stress field caused by the mismatch of CTEs between the matrix and the particulates. The role of thermal residual stress induced by CTE mismatch has been considered as a major cause for the crack deflection by Wei and Becker,<sup>5</sup> although no quantitative estimate on the increased toughness was made. In principle, quantitative analysis of toughening due to crack deflection by thermal residual stress can be made within the framework of the Fable-Evan's crack deflection model, but such an analysis would become intractable. Another toughening mechanism that has *not* been studied so far is the thermal residual stress in the *matrix domain*. If such residual stress in the matrix is to contribute to toughening, then the thermal residual stress in the matrix must be compressive. This negative thermal residual stress in the matrix can exist when the CTE of the particulate exceeds that of the matrix. This is indeed the case with the TiB2-particulate/SiC-matrix<sup>6,8,9</sup> and TiC-particulate/SiC-matrix composites.<sup>5</sup>

In order to quantify the influence of thermal residual stress, a combined experimental and theoretical analysis on the  $TiB_2/SiC$  composite as a model system was undertaken. Experimental analysis, followed by a brief description of the analytical model will be presented in Sections II and III, respectively. Finally, a comparison between the experimental and theoretical results will be presented in Section IV.

# II. Experiment

# 2.1 Materials

The ceramic matrix composite considered is a 16 percent volume  $TiB_2$  particulate/SiC matrix ( $TiB_2/SiC$ ) composite. The unreinforced SiC and  $TiB_2/SiC$ 

composite\* were processed by pressureless sintering in an inert atmosphere at a temperature in excess of 2000°C. The room temperature material properties of SiC matrix and TiB<sub>2</sub> particulate are given in Table 1.6,16,17

The temperature dependency of the CTE of SIC and  $TiB_2$  within the temperature range of 200 to  $2200K^{16,17}$  is given in Fig. 1, and is used in Section III to calculate the thermal residual stresses induced by the CTE mismatch between the matrix and the particulate.

Photographs of typical backscattered electron image (BEI) of as-processed monolithic SiC and TiB<sub>2</sub>/SiC composite are shown in Fig. 2(a) and (b), respectively. In Fig. 2(b), the isolated light gray areas and the continuous domain of gray are TiB<sub>2</sub> particulates and SiC matrix, respectively, where the dark isolated spots indicate voids. Voids are also seen in the unreinforced SiC as dark spots in Fig. 2(a). Some of the voids in Fig. 2(b) appear to be TiB<sub>2</sub> particulates, which fell out during polishing, due to weak bonding of the matrix-particulate.<sup>6</sup> A number of BEI photos similar to that of Fig. 2(b) were used to measure the average interparticulate spacing,  $\lambda$  (between the centers of two particulates), and the volume fraction of voids,  $f_V$  (based on ASTM Standard E562–76<sup>18</sup>). The results are  $\lambda = 11.1 \mu m$  and  $f_V = 0.016$ , which will be used in the analytical model. The value of  $f_V$  so measured is consistent with the density of the composite, i.e.,  $(1-f_V) \equiv 0.98$ –0.99 reported by McMurty et al.<sup>6</sup>

### 2.2 Measurement of Crack Growth Resistance, KR

The method of obtaining crack growth resistance,  $K_R$ , is based on a hybrid experimental-numerical analysis<sup>20</sup> where moire interferometry was used. The purpose of using moire interferometry was to measure the crack opening displacement (COD) along the crack as well as at the load point. A schematic of the moire interferometry system used in this study is shown in Fig. 3. A moire specimen with an argon laser of 514.5 nm wavelength, a grating, F, of 600 lines/mm and a virtual grating of 2F = 1200 lines/mm were used. The displacement, u, can be measured by counting the number of the moire fringe  $N^{1.9}$ 

$$u = \frac{N}{2F} \tag{1}$$

The specimen used is a chevron-notched, a wedge-loaded double-cantilever beam (WL-DCB) type, which is shown in Fig. 4 where the dimensions unit are in mm. A side groove with a depth of one half of the specimen thickness was used to propagate a straight crack along the crack plane (x-axis). A chevron notch was used to generate a true crack. The effect of the side groove on the stress intensity factor is minimal.<sup>21</sup> The moire interferometry grating, which consists of a thin aluminum film on an epoxy adhesive, is attached to the front side (not the groove side). The displacement along the

<sup>\*</sup> Supplied by British Petroleum Company, Niagara Falls, N.Y.

y-axis,  $u_y$ , on the front side is measured by the moire during loading. As shown in Fig. 4, the length of a two-dimensional crack, a, is measured from the intersection of the chevron notch with the front side (x=0, y=0). A typical moire fringe pattern of a TiB<sub>2</sub>/SiC composite specimen is shown in Fig. 5. The displacement,  $u_y$ , along the x-axis, i.e., the one-half crack opening displacement (COD) was measured by taking the average of the  $u_y$  values on the left and right side fringes since the fringe pattern is often unsymmetrical as shown in Fig. 5

For the hybrid analysis, the  $u_y$  at the load point, i.e., the contact point with the WL-DCB specimen, together with the measured vertical load,  $p_x$ , are input to a two-dimensional finite element code for computing the stress intensity factor,  $K_I$ . This procedure eliminates the uncertainty in numerical analysis associated with the friction at the loading pin-specimen contact. The boundary conditions and the pattern of the finite element analysis are shown in Fig. 6 where only one half of the WL-DCB specimen was used for the analysis. The results of the COD measured (symbols) and predicted by the finite element analysis (solid curves) are plotted as a function of x for various stages of loading in Fig. 7. This finite element analysis was validated by the excellent agreement between the computed and measured (by moire interferometry) CODs along the crack plane in Fig. 7. Finite element analysis was then used to compute the stress intensity factor using the standard strain energy release rate method. This procedure was repeated for each incremental increase in crack length,  $\Delta a$ , which then yielded the crack growth resistance  $K_B$ .

Crack growth resistance curves,  $K_R-\Delta a$ , were thus obtained for the monolithic SiC and  $TiB_2/SiC$  composites under monotonically increasing load as shown in Fig. 8(a), and cyclically increasing load as shown in Fig. 8(b). The resultant  $K_R-\Delta a$  relations are shown in Fig. 9 where the circles and the squares denote the monolithic SiC and  $TiB_2/SiC$  composites, respectively. The open and filled symbols represent the results of monotonic and cyclic loadings, respectively. Fig. 9 shows that  $K_R$  of the composite increased by 60 percent (cyclic loading)  $\sim$  80 percent (monotonic loading) over that of the monolithic SiC. Such increases in  $K_R$  cannot be predicted by the existing models alone, since the toughness increase predicted by the crack deflection model<sup>3</sup> is only 14 percent above the monolithic matrix and is much lower than that obtained experimentally.

### III. Analytical Model

The analytical modeling consists of two steps. The first step is to calculate the average thermal residual stresses in the matrix and reinforcement by using the Eshelby's  $model^{22}$  which is modified for a finite volume fraction of particulates.<sup>23–26</sup> The second step is to calculate the change in the mode I stress intensity factor,  $\Delta K$ , due to the compressive residual stress (obtained in the first step) in the matrix. In this model, the crack is assumed to propagate toward the nearest tensile stress region, which coincides with the domain surrounding the particulate generated by the thermal residual stress.

### 3.1 Thermal Residual Stress

A particulate composite is assumed to consist of three phases: spherical particulates of volume fraction  $f_p$ , spherical voids of volume fraction  $f_v$  and the matrix with its volume fraction of  $1-f_p-f_v$ . In order to calculate the thermal residual stress in a composite at room temperature,  $T_R$ , the composite is assumed to be subjected to a temperature change,  $\Delta T = T_R-T_P$  where  $T_P$  denotes the processing temperature such as sintering temperature. The thermal residual stresses in a composite are assumed to be induced by elastic deformations of the matrix and particulates under a uniform  $\Delta T$ . The voids are included since the SEM observation of Fig. 2 shows numerous voids distributed in a composite, despite the small volume fraction ~1.6 percent.

The modified Eshelby's model is suited for calculating the average internal stress field in a composite.  $^{26,27}$  The particulate composite containing voids subjected to uniform temperature change,  $\Delta T$ , is schematically shown in Fig. 10(a) where the domains of the particulates, voids, and the entire composite are denoted by  $\Omega_1$ ,  $\Omega_2$  and D respectively, thus the matrix domain becomes  $D-\Omega_1-\Omega_2$ . The elasticity problem of Fig. 10(a) can be reduced to that of Fig. 10(b) where the CTE's and  $\Delta T$  are converted to the CTE misfit strains,  $\alpha_1$  (in  $\alpha_1$ ) and  $\alpha_2$  (in  $\alpha_2$ ), which are given by

$$\alpha^{1} = \int_{T_{p}} (\alpha_{p} - \alpha_{m}) \delta dT$$
 (2)

$$g^2 = \int_{T_P}^{T_R} (\alpha_V - \alpha_m) \delta \ dT \tag{3}$$

and where  $\alpha_p$ ,  $\alpha_V$  and  $\alpha_m$  are the CTEs of the particulate, void and matrix, respectively, and  $\underline{\delta}$  is the isotropic tensor (Kronecker's delta). The subscripted tilda denotes a tensorial quantity.  $\alpha_V$  is not involved in the final formulation since the stiffness of  $\Omega_2$  vanishes.

The modified Eshelby's model Fig. 10(b), is equivalent to Fig. 10(c) where two inhomogeneities  $(\Omega_1,\Omega_2)$ , with the misfit strains defined by eqs. (2) and (3) are replaced by the inclusions with eigenstrains of  $e^1$  in  $\Omega_1$  and  $e^2$  in  $\Omega_2$ . The thermal stresses in a composite can then be solved in terms of the eigenstrains. Detailed formulation for the thermal stresses in a particulate composite containing voids is given in the Appendix. Only the average thermal stresses, which will be used later in this section for calculations of the stress intensity factor change,  $\Delta K$ , will be considered. The average stress field in the particulate  $\langle \sigma \rangle_p$  and in the matrix  $\langle \sigma \rangle_m$  are isotropic since the particulate reinforcement and the CTE misfit strains are dilatational. From the Appendix, these are

$$\frac{\langle \sigma \rangle_{p}}{E_{m}} = \frac{-2(1-f_{p}-f_{v})\beta\alpha_{1}}{A}$$
 (4)

$$\frac{\langle \sigma \rangle_{m}}{E_{m}} = \frac{2f_{p}\beta \alpha_{1}^{*}}{A}$$
 (5)

where

$$A = (1-f_{V}) \left[ \left\{ 1 - \frac{f_{D}}{(1-f_{V})} \right\} (\beta+2)(1+v_{m}) + \frac{3\beta f_{D}(1-v_{m})}{(1-f_{V})} \right]$$

$$\beta = \left( \frac{1+v_{m}}{1-2v_{D}} \right) \left( \frac{E_{D}}{E_{m}} \right)$$
(6)

 $f_p$  and  $f_v$  are the volume fraction of the particulates and voids, respectively.  $E_i$  and  $v_i$  are the Young's modulus and Poisson's ratio of the i-th phase, where i = m and p represent the matrix and particulate, respectively. When the composite does not contain voids, the above equations can be simplified as

$$\frac{\langle \sigma \rangle_{p}}{E_{m}} = \frac{-2(1-f_{p})\beta \alpha_{1}^{*}}{A} \tag{7}$$

$$\frac{\langle \sigma \rangle_{m}}{E_{m}} = \frac{2f_{p}\beta \alpha_{1}^{*}}{A}$$
 (8)

where

$$A = (1-f_p)(\beta+2)(1+v_m) + 3\beta f_p(1-v_m)$$
 (9)

It is clear from the above equations that the thermal residual stresses are influenced by a number of factors: the particulate-matrix stiffness ratio; the Poisson's ratios of the matrix and particulate; the volume fractions of the particles and voids; and the CTE misfit strain,  $(\alpha_p - \alpha_m)\Delta T$ . The formula used by Wei and Becker does not contain the volume fraction of the particulates,  $f_p$ , thus it is a lower order approximation to the stresses in a composite with a finite volume fraction of particulates.

For a particulate reinforced ceramic matrix composite with  $\alpha_p > \alpha_m$ , such as a TiB<sub>2</sub>/SiC composite,  $\alpha_1^* < 0$  as seen from eqs. (5) and (6) the average thermal stresses in the particulates and matrix are in tension and compression, respectively. The thermal residual stress field in a TiB<sub>2</sub>/SiC composite consists of two regions: the tensile stress region in the particulates and their vicinity in the matrix, and the compressive stress region in the bulk of the matrix, as shown schematically in Fig. 11.

# 3.2 Stress Intensity Factor Reduction Due to Thermal Residual Stress in Matrix

Consider a semi-infinite crack surrounded by a particulate-reinforced/ceramic-matrix composite with a thermal residual stress distribution as shown in Fig. 11. The semi-infinite crack is assumed to advance from one concentrated tensile region to its

nearest counterpart which may be off the crack plane. Thus, the crack may be deflected according to Wei and Becher,<sup>5</sup> but the general crack path over a number of tensile regions will be that of a self-similar crack extension. Hence, a self-similar crack extension over an average interparticulate spacing,  $\lambda$  will be considered. When a semi-infinite crack advances by a distance  $\lambda$ , which is the interparticulate distance, the crack tip ligament is subjected to the local average compressive stress, q, over the length of  $\lambda$ -d, as shown in Fig. 12, in addition to the superposed tensile field for self-similar crack propagation. "d" denotes the average diameter of TiB<sub>2</sub> particles. The existence of this local stress, q, decreases the stress intensity factor,  $\Delta$ K<sub>1</sub>. According to Tada, <sup>28</sup> this decrease is

$$\Delta K_{\parallel} = \frac{2}{\pi} q \sqrt{2\pi(\lambda - d)}$$
 (10)

# IV. Results and Discussion

#### 4.1 Results

Using the analytical model developed in the preceding section, the increase in the fracture resistance of a TiB $_2$  particulate-reinforced/SiC-matrix composite will be predicted. The analysis involves two steps: calculation of the average thermal residual stresses; and  $\Delta K_L$ 

The formulae for calculating the average thermal stresses in the particle,  $\langle \sigma \rangle_p$ , and the matrix,  $\langle \sigma \rangle_m$ , are given by eqs. (4) and (5), or eqs. (7) and (8) for small values of  $f_V$ , respectively. The parameters A,  $\beta$  and  $\alpha_l$  must be calculated first using known input  $f_p$ ,  $f_V$ ,  $v_m$ ,  $v_p$ ,  $E_p$  and  $E_m$ . Since the measured volume fraction of voids is very small, i.e.,  $f_V = 0.016$ , eqs. (7) and (8) will be used in this analysis.

The value of the CTE mismatch strain,  $\alpha_1$ , is defined by eq. (2). The CTE's of the matrix (SiC),  $\alpha_m$ , and the TiB<sub>2</sub> particulate,  $\alpha_p$ , are given as a function of temperature T(K) in Fig. 1. By taking  $T_R = 300 K$  and  $T_P = 2273 K$  and integrating  $\alpha_p - \alpha_m$  with respect to T over the entire temperature range from  $T_P$  to  $T_R$ , eq. (2) yields

$$\alpha_1 = -5.15 \times 10^{-3} \tag{11}$$

Parameters  $\beta$  and A are calculated using the data in Table 1 and are

$$\beta = 3.50$$
 (12)  $A = 6.86$ 

where  $f_V = 0$  was assumed. From eqs. (7), (8), (11) and (12) and using  $f_p = 0.16$ , the average thermal residual stresses are computed as

$$\langle \sigma \rangle_{m} = -345 \text{ MPa}$$

$$\langle \sigma \rangle_{p} = 1810 \text{ MPa}$$
(13)

Next an estimate of the toughness increase due to the thermal residual stress in the matrix is made. In eq. (10), we set

$$q = \langle \sigma \rangle_m = -345 \text{ MPa}$$

$$\lambda = 11.1 \mu m$$

$$d = 3.06 \mu m$$
(14)

where  $\lambda$  is the average interparticulate spacing which was obtained from ten SEM photos containing 70 TiB<sub>2</sub> particulates. Substituting eq. (14) into (10) yields

$$\Delta K_{l} = -1.56 \text{ MPa} \tag{15}$$

The compressive residual stress in the matrix thus reduces the stress intensity factor by 1.56 MPa. This reduction in  $\Delta K_{I}$  is equivalent to the increase in the crack growth resistance  $\Delta K_{B}$  by the same amount. Hence

$$\Delta K_{R} = 1.56 \text{ MPa} \tag{16}$$

### 4.2 Discussion

The measured and predicted increases in fracture resistance is now compared. The measured increase in  $K_R$ ,  $\Delta K_R$ , can be obtained from Fig. 9 for the case of monotonic loading,

$$\Delta K_R = (K_R)$$
 composite  $-(K_R)$  monolith  
=  $(5.3 - 3.0)$  MPa  $\sqrt{m}$  (17)  
=  $2.3$  MPa  $\sqrt{m}$ 

It is clear from eqs. (16) and (17) that the major portion of  $\Delta K_R$  is attributed to toughening due to the thermal residual stress. If we take the toughness ratio between the composite,  $K_C$ , and the monolithic matrix,  $K_R$ , the measured and predicted values are

$$\frac{K_{c}}{K_{m}} = \begin{cases} 1.77 & \text{measured} \\ 1.52 & \text{predicted} \end{cases}$$
 (18)

The measured ratio is comparable to those reported by others.<sup>6,8</sup> The crack deflection model of Faber and Evans<sup>3</sup> predicts a K<sub>c</sub>/K<sub>m</sub> ratio of 1.14 for uniformly distributed TiB<sub>2</sub> particulates. Thus, the crack deflection model cannot account fully for the toughening of a ceramic matrix composite.

The predicted value of  $K_c/K_m=1.52$  is based on the assumption that  $T_P=2273$  K which is close to the sintering temperature of the composite and also that both constituent phases of SiC and TiB<sub>2</sub> deform elastically during the cool down process from 2273K to 300K. SiC  $\alpha$ -phase matrix is less susceptible to creep than other ceramics at a given high temperature due to its strong covalent bond and ionicity. Its activation energy for self diffusion is large and results in a slow diffusion rate and thus slow creep-rate.<sup>29</sup> This slow creep-rate gives rise to small stress relaxation. It is thus more realistic to assume that some stress relaxation due to the diffusion takes place during the high temperature regime. If  $T=1500^{\circ}$ C is considered as a threshold temperature,  $T_{th}$ , above which the stress relaxation due to the diffusion can occur, then  $T_P$  must be set equal to  $1500^{\circ}$ C in the elastic analysis of the thermal residual stress. For  $T_P=1500^{\circ}$ C, the predicted value of  $K_c/K_m=1.43$ . The toughening effect based on  $T_P=1500^{\circ}$ C still constitutes the major portion of the observed toughness increase. The threshold temperature  $T_{th}$  is, however, difficult to determine since the creep (diffusion) behavior is a continuous function of temperature and time.

 $T_{th}$  of metal matrix composites can be estimated by *in situ* transmission electron microscopy (TEM) study<sup>30</sup> as the temperature, at which the dislocations start to appear in the matrix close to the matrix-filler interface during the cool down process. Such an *in situ* TEM study of a ceramic composite is difficult to conduct since  $T_{th}$  is too high for heating stage device within a TEM. In addition, the generation (punching) of the dislocations from the matrix-filler interface due to the CTE mismatch<sup>31</sup> is unlikely due to high friction stress of the ceramics matrix.

It was assumed in the present model that the crack propagation path over a number of tensile regions, each surrounding a particulate, is self-similar despite the locally wavy fracture surface. Figure 13(a) shows a typical BEI photo of a crack propagating in a  $TiB_2$  particulate-reinforced/SiC-matrix composite toward the right and Fig. 13(b) is a portion of Fig. 13(a) at higher magnification . It is noted in Fig. 13(b) that the dark regions on the crack path were occupied by  $TiB_2$  particulates which fell off during polishing. Both figures indicate that the crack actually propagated from particulate to particulate located on the crack plane. Figures 14(a) and (b) are BEI photos of the fracture surface of the composite and monolithic SiC, respectively. A comparison between Figs. 14(a) and (b) reveals that the degree of the local waviness of these photos is the same except for the regions next to particulates. This secondary wavy surface which is associated with the particulates are probably due to the crack deflection proposed by Faber and Evans<sup>3</sup>. The toughening due to this crack deflection was evaluated as  $K_c/K_m = 1.14$ . By superposing the toughening due to crack deflection onto that due to compressive stress in the matrix, one can estimate the toughness ratio as

$$\frac{K_{\rm C}}{K_{\rm m}} = 1 + 0.14 + 0.52 = 1.66 \tag{19}$$

The toughness ratio so estimated is close to the experimental value of  $K_0/K_m = 1.77$ .

The validity of the present model can be partially supported by the experimental results by Jenkins et al.<sup>9</sup> where the crack growth resistance,  $K_R$ , of  $TiB_2/SiC$  composite measured at various temperatures from room to  $1400^{\circ}C$  exhibited a decrease with increasing testing temperature. Toughening in the present model is proportional to the compressive thermal residual stress in the matrix, which in turn is proportional to the temperature change,  $\Delta T = T_T - T_P$ , where  $T_T$  is the testing temperature for measuring  $K_R$ . Hence, the higher testing temperatures result in smaller increase in  $K_R$  as observed by Jenkins et al.<sup>9</sup>

The effect of volume fraction of particulate,  $f_p$ , can be examined by looking at eqs. (7), (9) and (10). The thermal residual stress in the matrix  $\langle \sigma \rangle_m$  is an increasing function of  $f_p$ , as shown by eqs. (7) and (9), and will influence linearly  $\Delta K_l$  obtained by eq. (10). On the other hand, the average interparticulate spacing,  $\lambda$ , is proportional to  $1/\sqrt{f_p^3}$ 

$$\lambda = 1.085 \text{ d/}\sqrt{f_p} \tag{20}$$

while  $\Delta K_1$  is proportional to  $\sqrt{\lambda}$ -d, as shown by eq. (10). Using the average measured diameter of the particulates,  $d = 3.06 \mu m$ , one can calculate the interparticulate distance.  $\lambda$ , by eq. (20), which yields  $\lambda = 8.14 \ \mu m$ . This value of  $\lambda$  is smaller than the average of the measured interparticulate distances on 70 TiB<sub>2</sub> particulates,  $\lambda$  = 11.1  $\mu$ m. This discrepancy between the measured and that predicted by eq. (20) is attributed to some error involved in our measurement or the degree of approximation inherent in eq. (20). Non-uniform distribution of TiB<sub>2</sub> particulate also contributed to a larger measured  $\lambda$  is larger than that predicted by eq. (20). By combining the above effects, the toughness ratios,  $K_c/K_m$ , are computed for various values of  $f_p$ . The predicted results of the  $K_c/K_m$ versus for relation are shown as a solid curve in Fig. 15, with the experimental value at for = 0.16 shown as an open circle. Also shown in Fig. 15 are the prediction of KR based on the crack deflection model by Faber and Evans for two cases of particulates distribution; uniform spacing (dash) and distributed spacing (dash-dot). It is clearly seen from Fig. 15 that the present model can predict the observed toughness increase better than the crack deflection model and it can account for the major toughening of a composite. It follows from Fig. 15 that the crack growth rate ratio of the composite to the monolithic matrix increases with  $f_p$ . The rate of increase in  $K_c/K_m$  is, however, not linear with  $f_p$  but decreases with increase in fp. This fp dependence of the fracture toughness ratio has also been observed by other investigators. 6,33 As fp increases to higher values, for example, in excess of 0.5, the present model fails to predict the correct trend of the Kc/Km versus fp relation. At such high volume fractions, many particulates come in contact and form large clusters. The microstructure of these clusters differs from that of the present model, as shown in Fig. 10. Thus, the K<sub>c</sub>/K<sub>m</sub> ratio would be saturated, or even start to decrease at higher volume fractions. Endo et al.<sup>33</sup> have indeed observed such a trend with

several non-oxide particulates-reinforced/SiC-matrix composite systems. Figure 16 shows the  $K_{\rm IC}$  versus  $f_{\rm D}$  relation of TiC particulate/SiC matrix composite.

The present model predicts high tensile stress in TiB2 particulate at room temperature as per eq. (13). If this high tensile stress  $<\sigma>p$  exceeds the bonding strength of the matrix-particulate interface,  $\sigma_b$ , then the interface will be debonded. It seems appropriate to assume that the interfacial bonding strength varies from particulate to particulate. Thus, some particulates with  $\sigma_b \le \langle \sigma \rangle_p$  would become debonded voids where the debonded interface cannot transfer stress except for compressive stress. however, difficult to determine the percentage of such particulates with weak interface, but one can obtain a rough estimate of the percentage by observing the percentage of TiB2 particulates that would fall out from the specimen during the polishing. If the debonded voids so nucleated are also counted, then the volume fraction of voids, fy, increases as the fraction of TiB2 particulates with weak interface increases. The effect of fy on KR can be studied by calculating the average compressive stress in the matrix  $\langle \sigma \rangle_m$  using eqs. (5) and (6). The solid curve in Fig. 17 shows the results of  $\langle \sigma \rangle_m$  as a function of  $f_V$  by assuming that voids are nucleated only from TiB<sub>2</sub> particulates, i.e.,  $f_V + f_D = 0.16$ .  $\langle \sigma \rangle_m$  is found to decrease with fy and becomes zero when all the TiB2 particulates are changed to Thus the voids nucleate as a result of the debonding of the matrix-particulate interface would reduce the toughness of a particulate composite. On the other hand, if we assume that the voids are induced as a result of the processing (sintering and cool down process), and the matrix-particulate interfaces are bonded, then fy dependence of  $<\sigma>_m$ , as shown by the dashed curve in Fig. 17, has the opposite trend to the above case. Namely,  $<\sigma>_m$  increases gradually with the increase in  $f_V$ , thus contributing to the toughness of a particulate composite, although the increase in the toughness is modest. The values of fv. however, are usually small as in the present case ( $f_V = 0.016$ ). Thus the effect of voids on KR can be considered minimal.

The effect of cyclic loading on  $K_R$  of a TiB<sub>2</sub> particulate-reinforced/SiC-matrix is obvious from Fig. 9. Namely, the cyclic loading defined by Fig. 8 tends to reduce the value of  $K_R$  by about 0.7 m $\sqrt{Pa}$ . This reduction in  $K_R$  can be explained as follows: the cyclic loading modifies the local stress field including the residual thermal stress, resulting in the lower compressive stress in the matrix. Thus a crack front is subjected to such a modified thermal stress field during the loading process following the preceding unloading, leading to the reduction in the toughness. This modified thermal stress field would not occur in the monolithic SiC due to the lack of TiB<sub>2</sub> particulates.

### IV. Conclusion

Increase in the K<sub>R</sub> of the composite over the unreinforced matrix is attributed to new analytical model based on the compressive thermal residual stress in the matrix in a particulate reinforced ceramic matrix composite. The K<sub>R</sub> predicted by this model agrees with the experimental value better than an existing model.

### V. Acknowledgements

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Table 1 Material Properties of SiC and TiB<sub>2</sub>

Properties	Unit	SIC	TiB <sub>2</sub>
Crystal structure		hexagonal	hexagonal
Specific gravity	g/cm <sup>3</sup>	3.21	4.52
Melting point	℃	2540°	2790
Young's modulus	GPa	410	531
Poisson's ratio	1	0.19	0.28
Average size	μm	grain size 3	2.06***
Volume fraction		0.84	0.16
CTE"	10-6/°C	4.02	4.6

<sup>\*</sup> Degassing temperature

<sup>\*\*</sup> These CTE values taken from ref. 6 are averages over room temperature~700°C range, and are not accurate. The accurate CTE values of SiC and TiB<sub>2</sub> 200~2200K<sup>16,17</sup> are given in Fig. 1.

<sup>\*\*\*</sup>This value, which is taken from ref. 6, is smaller than the average value that we have measured. This is discussed later.

# Appendix: The Formulation for the Thermal Residual Stresses

Referring to Fig. 10(b), the internal elastic stress field in domain  $\Omega_1$  (particulates) and  $\Omega_2$  (voids) are denoted by  $g^1$  and  $g^2$ , respectively, which are:

$$g^{1} = C^{p} \cdot (\overline{e} + e^{1} - g^{1}) \quad \text{in } \Omega_{1}$$
 (A1)

$$g^2 = C^{\vee} \cdot (\overline{e} + e^2 - g^2)$$
 in  $\Omega_2$  (A2)

where the bold face letters and those with subscripted tildas denote tensorial quantities,  $C^p$  and  $C^v$  are the stiffness tensor (of rank 4) of the particulate and void, respectively,  $e^1$  and  $e^2$  are the strain disturbed by  $\Omega_1$  and  $\Omega_2$ , respectively, and  $\overline{e}$  is the average strain in the matrix, and related to the average stress in the matrix  $\langle \sigma \rangle_m$  as

$$\langle \sigma \rangle_{\mathsf{m}} = \mathsf{C}^{\mathsf{m}} \cdot \overline{\mathsf{e}} \tag{A3}$$

The dots used in the above equations denote inner product between tensors. Following Eshelby,<sup>22</sup> Fig. 10(b) can be further reduced to Fig. 10(c) where two inhomogeneities, particulates and voids, are now replaced by two inclusions. An inclusion is a domain surrounded by the matrix and possesses the same properties as the matrix and also an eigenstrain e\* which is an inelastic strain.<sup>24</sup> Thus Equations (A1) and (A2), which are formulated based on Fig. 10(b), are now reduced to the following, Fig. 10(c):

$$\underline{\sigma}^1 = \mathbf{C}^m \cdot (\overline{\mathbf{e}} + \mathbf{e}^1 - \mathbf{e}^{*1}) \quad \text{in } \Omega_1$$
 (A4)

$$g^2 = C^m \cdot (\overline{e} + e^2 - e^{*2}) \quad \text{in } \Omega_2$$
 (A5)

Here eqs. (A4) and (A5) are valid for the entire composite domain, D, if the eigenstrains,  $e^{*1}$  and  $e^{*2}$ , are defined as zero in the domain outside  $\Omega_1$  and  $\Omega_2$ , respectively. Noting that the integration of the stress field  $\sigma_1$  and  $\sigma_2$  over D vanishes:

$$\int_{0}^{\infty} dv = 0 \tag{A6}$$

we obtain

$$\bar{\mathbf{e}} = -f_p (\mathbf{e}^1 - \mathbf{e}^{*1}) - f_v (\mathbf{e}^2 - \mathbf{e}^{*2})$$
 (A7)

where  $f_p$  and  $f_v$  are the volume fraction of the particulate and void, respectively. The strain field,  $e^1$  and  $e^2$ , in the inclusions, are related to the corresponding eigenstrain,  $e^{*1}$  and  $e^{*2}$ , respectively, by the Eshelby's tensor S:

$$e^1 = S \cdot e^{1} \tag{A8}$$

$$e^2 = S \cdot e^{2} \tag{A9}$$

Eshelby's tensor (of rank 4) for an isotropic matrix is a function of the Poisson ratio of the matrix,  $v_m$ , and the geometry of  $\Omega$ .<sup>22,24</sup>

By equating eqs. (A1) and (A2) to eqs. (A4) and (A5), using  $C^{\vee} = 0$  due to voids, and eqs. (A7) through (A9), we arrive at:

$$C^{p} \cdot [(1-f_{p})S \cdot e^{1} + f_{p}e^{1} - f_{v}(S \cdot e^{2} - e^{2}) - g^{1}] =$$

$$C^{m} \cdot [(1-f_{p})(S \cdot e^{1} - e^{1}) - f_{v}(S \cdot e^{2} - e^{2})]$$
 (A10)

$$-f_{p}e^{*1} + (1 - f_{v})e^{*2} = 0$$
 (A11)

where the isotropic stiffness tensors,  $C^m$  and  $C^p$ , and misfit strain  $g^{*1}$  are given in index form as:

$$C_{iikl}^{J} = \lambda_{J}\delta_{ij}\delta_{kl} + \mu_{J}(\delta_{ik}\delta_{jl} + \delta_{il}\delta_{kj}), \quad J = m \text{ or } p$$
(A12)

$$\alpha_{ij}^{\uparrow 1} = \int_{T_P}^{T_R} (\alpha_p - \alpha_m) \, \delta_{ij} \, dT \qquad (A13)$$

where  $\delta_{ij}$  is the Kronecker's delta,  $\lambda_J$  and  $\mu_J$  are Lamé constants of the J-th phase material (J = m for the matrix, J = p for the particulate),  $\alpha_m$  and  $\alpha_p$  are the CTE of the matrix and particulate, respectively, and  $T_R$  and  $T_P$  are the room and processing temperatures, respectively.

The problem, which is illustrated Fig. 10(c), gives rise to isotropic state since the particulate reinforcement and the CTE mismatch strain are dilatational. Hence only one independent component for each unknown eigenstrain in eqs. (A10) and (A11) exists. Eshelby's tensor components required for solving eqs. (A10) and (A11), thus are:

$$S_{1111} = S_{2222} = S_{3333} = \frac{7 - 5v_m}{15(1 - v_m)}$$

$$S_{1122} = S_{2233} = S_{3311} = S_{1133} = S_{3322} = S_{2211} = \frac{5v_m - 1}{15(1 - v_m)}$$
(A14)

After substituting eq. (A14) into eqs. (A10) and (A11) and eliminating  $e^{*2}$ , we can solve for  $e_1 = e_{11} = e_{22} = e_{33}$ 

$$e_{1}^{*} = \frac{3\beta\alpha_{1}^{*}}{\left[\left\{1 - \frac{f_{p}}{(1-f_{v})}\right\}(\beta+2)\left(\frac{1+v_{m}}{1-v_{m}}\right) + \frac{3\beta f_{p}}{(1-f_{v})}\right]}$$
(A15)

where

$$\beta = \frac{3\lambda_p + 2\mu_p}{2\mu_m} = \frac{(1+\nu_m)}{(1-2\nu_p)} \left(\frac{E_p}{E_m}\right) \tag{A16}$$

The eigenstrain in  $\Omega_2$ ,  $e^{*2}$  (i.e.  $e_2 = e_{11}^{*2} = e_{22}^{*2} = e_{33}^{*2}$ ) can be obtained from eq. (A11) once  $e_1$  is solved.

The average stresses in the particulate  $\langle \sigma \rangle_p$  and in the matrix  $\langle \sigma \rangle_m$  are related to each other by eq. (A6).

$$f_p \langle \sigma \rangle_p + (1 - f_p - f_v) \langle \sigma \rangle_m = 0$$
 (A17)

where the stress in a void being zero was used, and  $\{\sigma\}_J$  with J=m (matrix) or p (particulate) denote an isotropic component, not tensor,  $\sigma$ 

The average stress in the particulate  $\langle \sigma \rangle_p$  can be calculated from eqs. (A4) and (A7) through (A9) once  $e_i$  and  $e_2$  are solved, and is given by

$$\frac{\langle \sigma \rangle_{p}}{E_{m}} = \frac{-2(1-f_{p}-f_{v})\beta\alpha_{l}}{(1-f_{v})\left[\left\{1-\frac{f_{p}}{1-f_{v}}\right\}(\beta+2)(1+\nu_{m}) + \frac{3\beta f_{p}(1-\nu_{m})}{(1-f_{v})}\right]}$$
(A18)

The average stress in the matrix  $\langle \sigma \rangle_m$  then is obtained from eqs. (A17) and (A18)

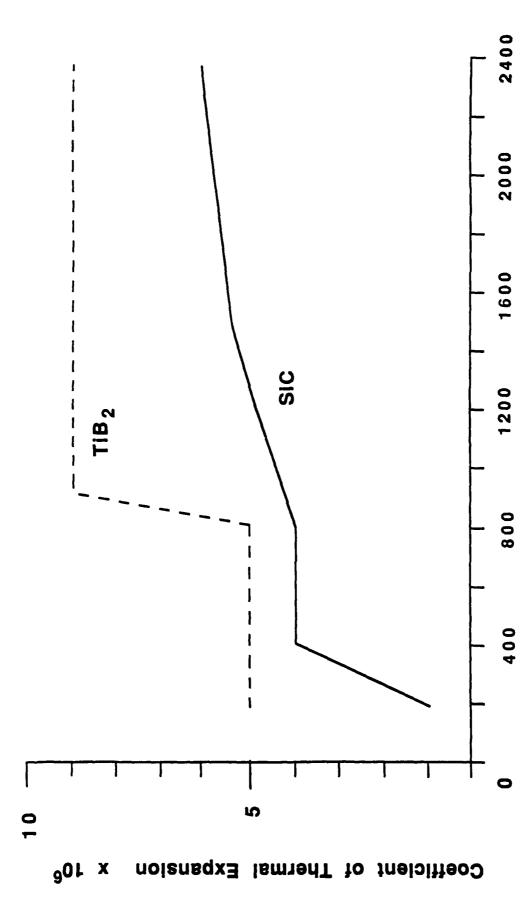
$$\frac{\langle \sigma \rangle_{m}}{E_{m}} = \frac{2f_{p}\beta\alpha_{1}^{2}}{(1-f_{v})\left[\left\{1-\frac{f_{p}}{1-f_{v}}\right\}(\beta+2)(1+v_{m}) + \frac{3\beta f_{p}(1-v_{m})}{(1-f_{v})}\right]}$$
(A19)

Eqs. (A18) and (A19) provide the average thermal residual stresses in a particle composite with voids due to CTE mismatch. When the composite does not contain voids,  $f_v = 0$ , eqs. (A18) and (A19) are simplified to

$$\frac{\langle \sigma \rangle_{p}}{E_{m}} = \frac{-2(1-f_{p})\beta\alpha_{1}}{\left[(1-f_{p})(\beta+2)(1+v_{m}) + 3\beta f_{p}(1-v_{m})\right]}$$
(A20)

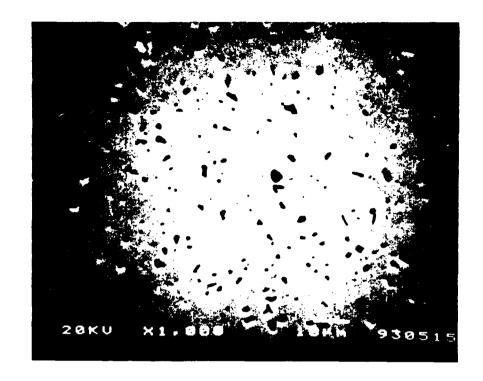
and

$$\frac{\langle \sigma \rangle_{m}}{E_{m}} = \frac{2f_{p}\beta\alpha_{1}^{2}}{\left[(1-f_{p})(\beta+2)(1+\nu_{m}) + 3\beta f_{p}(1-\nu_{m})\right]}$$
(A21)



Coefficients of thermal expansion of TiB2 and  $\alpha\textsc{-SiC}$  over the temperature range of 300-2400K. Fig. 1

Temperature (K)



(a)

(b)



Fig. 2 Typical backscattered electron image of: (a) as-processed SiC; and (b) TiB<sub>2</sub>/SiC composite.

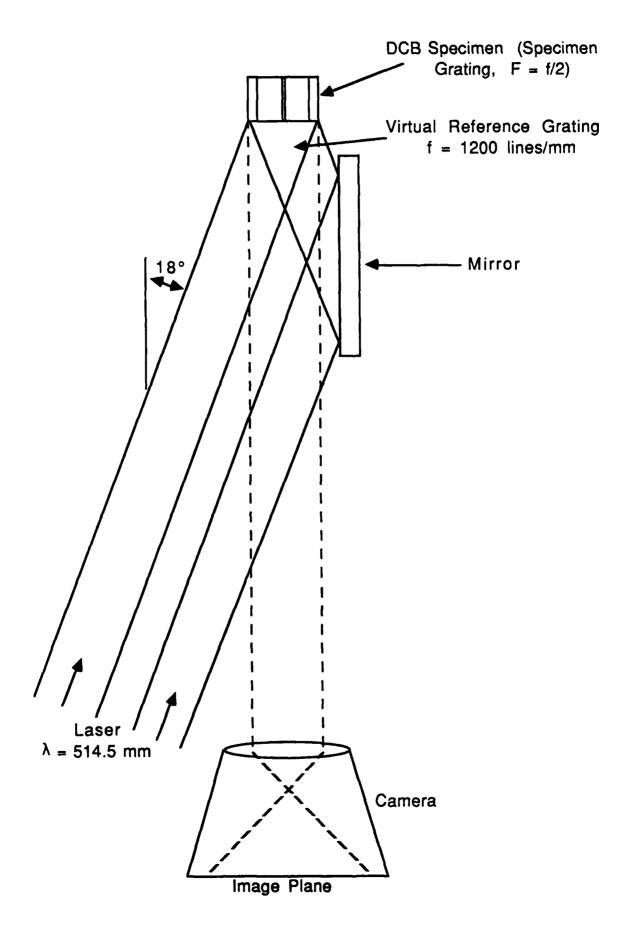
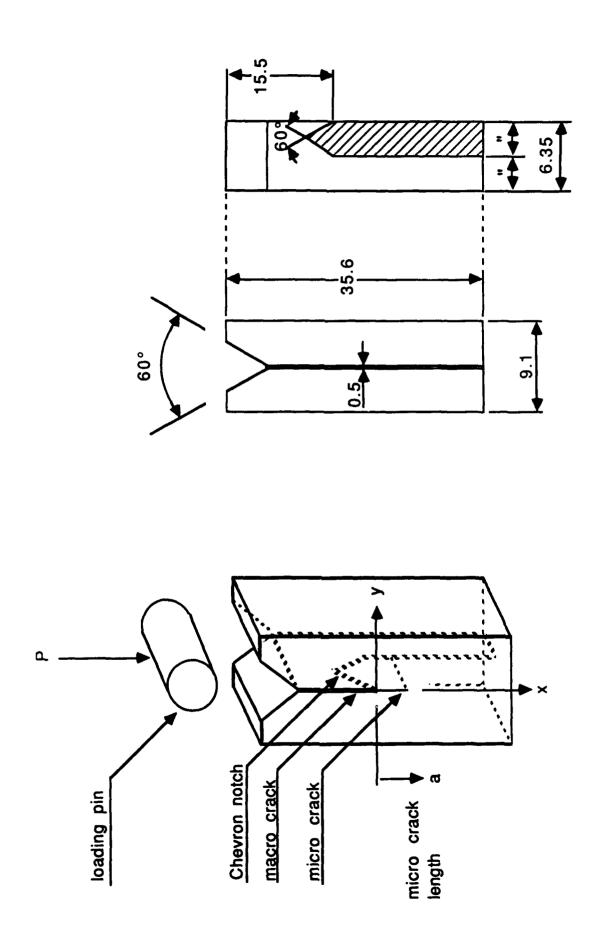


Fig. 3 Moire interferometry setup



Wedge-loaded double cantilever beam (WL-DCB) specimen with chevron notch and loading pin. All dimensions in mm. Fig. 4

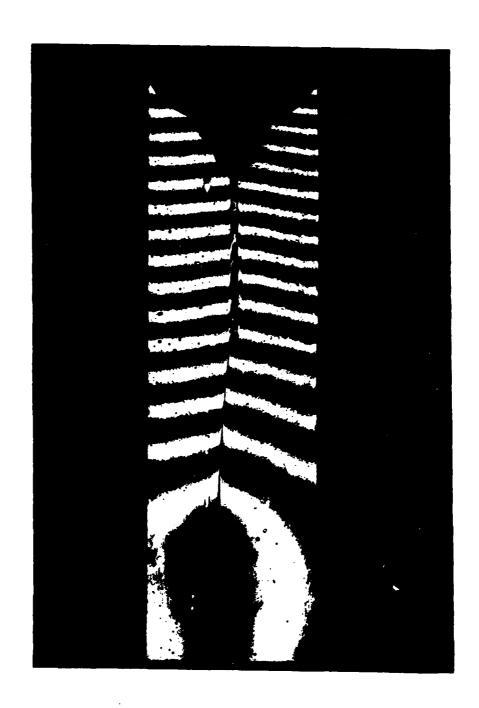


Fig. 5 A typical moire fringe pattern on a  $TiB_2/SiC$  composite specimen loaded to p=65 N with the corresponding crack extension  $\Delta a=9.6$  mm.

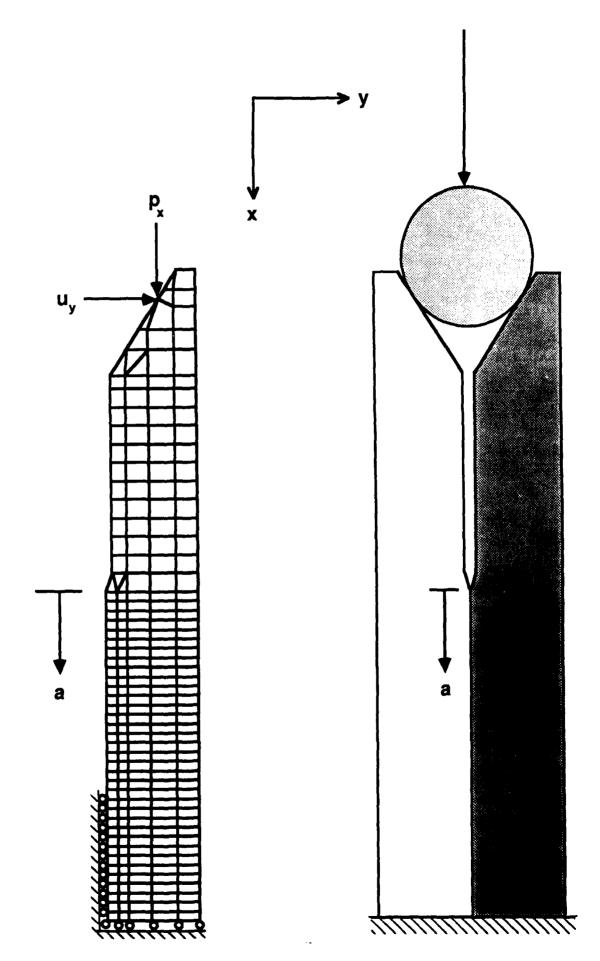
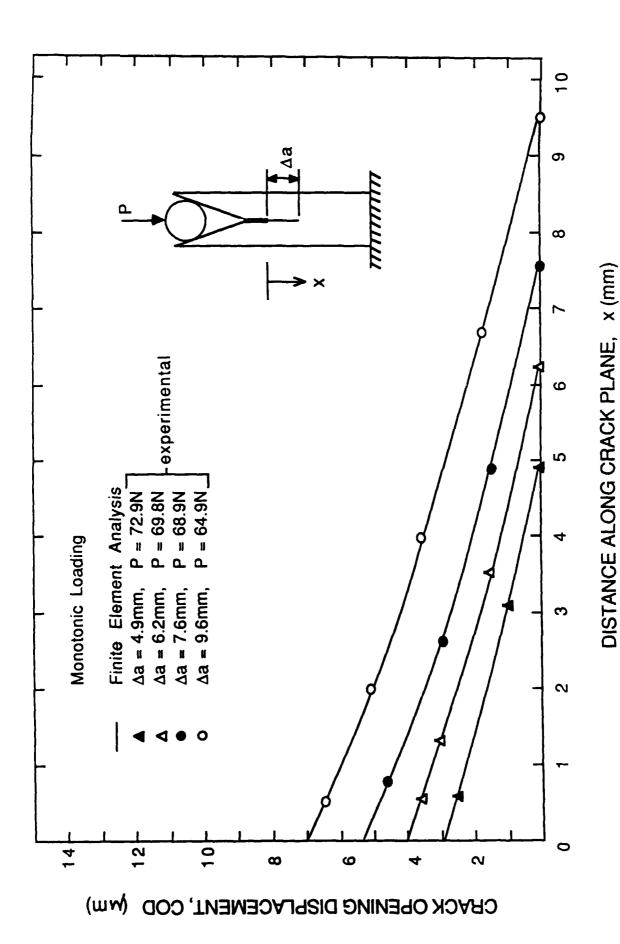
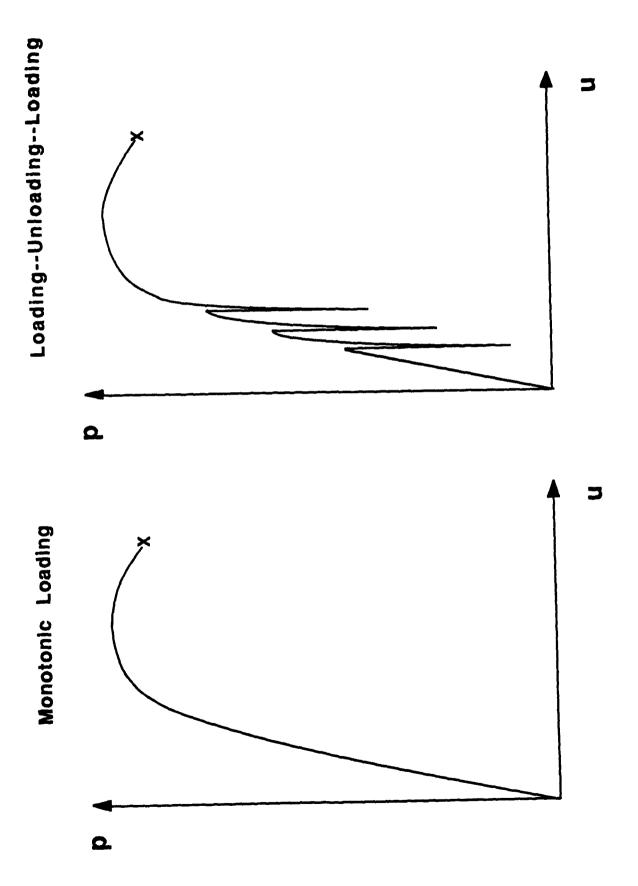


Fig. 6 2-D finite element analysis model with boundary conditions.



COD along crack plane for various crack extensions; finite element analysis (solid curve) and experimental results (symbols). Fig. 7



Load (p) versus displacement (u) relations; (a) monotonic and (b) cyclic.

Fig. 8

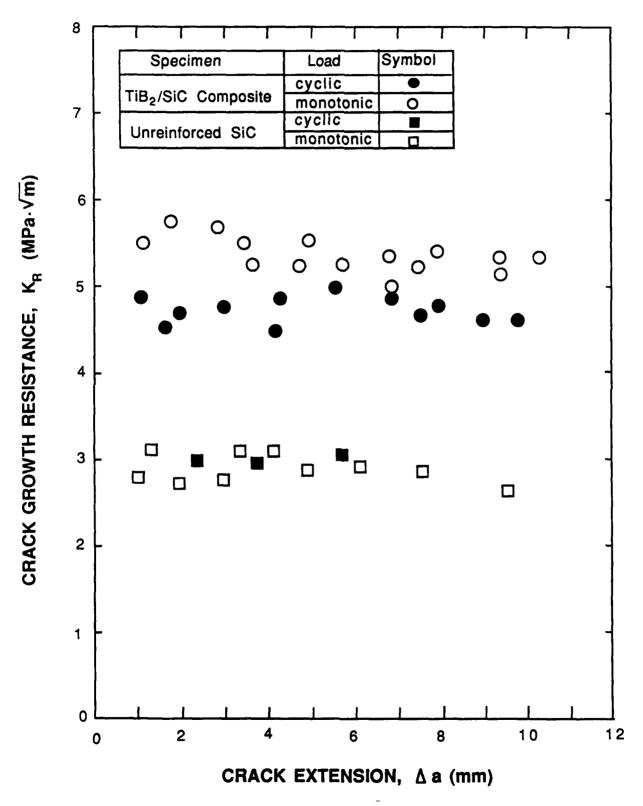


Fig. 9 Crack growth resistance,  $K_R$  versus crack extension,  $\Delta a$ , relations of the monolithic SiC (squares) and TiB<sub>2</sub>/SiC composite (circles).

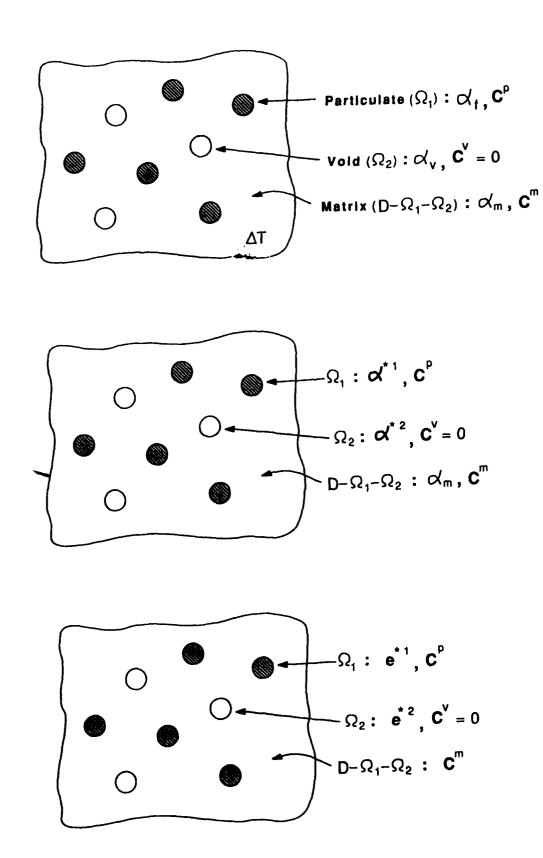


Fig. 10 Analytical model: (a) particulate reinforced composite with voids subjected to uniform temperature change  $\Delta T$ ; (b) two inhomogeneities with CTE misfit strains  $\alpha^{*1}$  in  $\Omega_1$  and  $\alpha^{*2}$  in  $\Omega_2$  embedded in the matrix  $D-\Omega_1-\Omega_2$ ; and (c) converted to the Eshelby's inclusion problem.

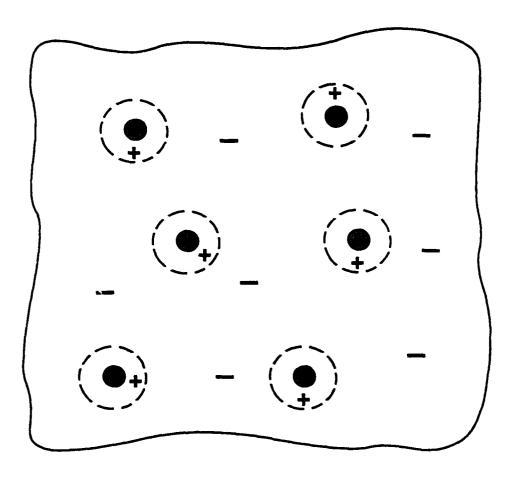


Fig. 11 Thermal residual stress field in a particulate-reinforced/ceramic-matrix composite.

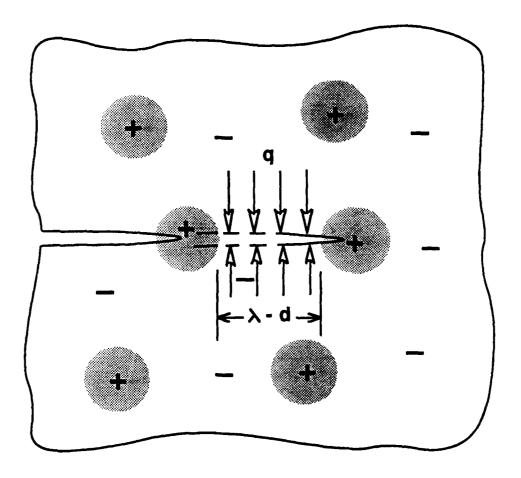


Fig. 12 Semi-infinite crack advances,  $\lambda$ , through matrix compressive region toward particulate tensile region.

30KII X750 10Hm. 950106

(a)

(b)

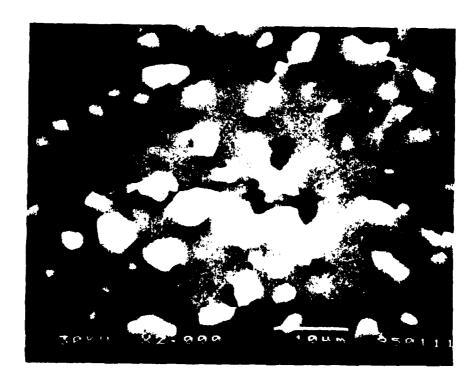
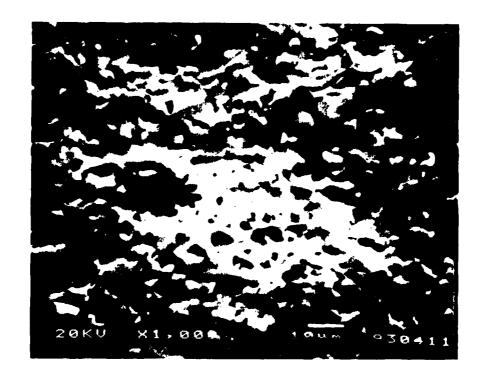


Fig. 13 (a) Backscattered electron image (BEI) of typical crack propagating path in a TiB<sub>2</sub> particulate-reinforced/SiC-matrix composite, (b) a segment of (a) at higher magnification.



(a)

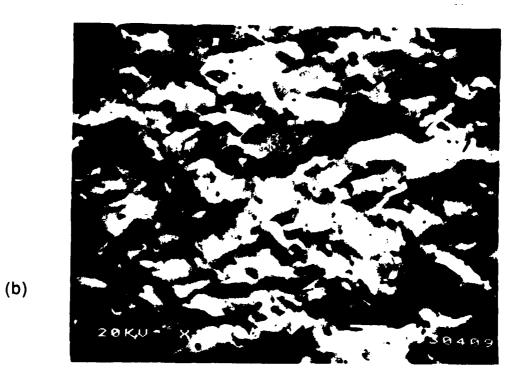


Fig. 14 BEI of fracture surface: (a) TiB<sub>2</sub>/SiC composite; (b) monolithic SiC.

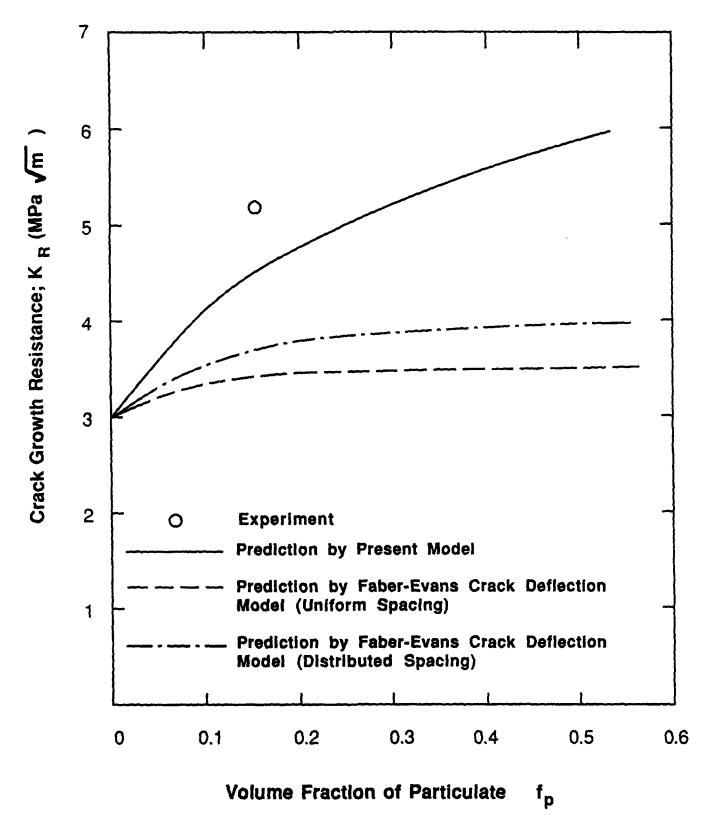


Fig. 15 The crack growth resistance ratio;  $TiB_2$ -particulate/SiC-matrix composite to the monolithic SiC, as a function of the volume fraction of  $TiB_2$  particulates,  $f_p$ . Predictions based on the present model (solid curve) and Faber-Evans crack deflection model (dash and dash-dot curves), and the present experimental result at  $f_p = 0.16$  (open circle).

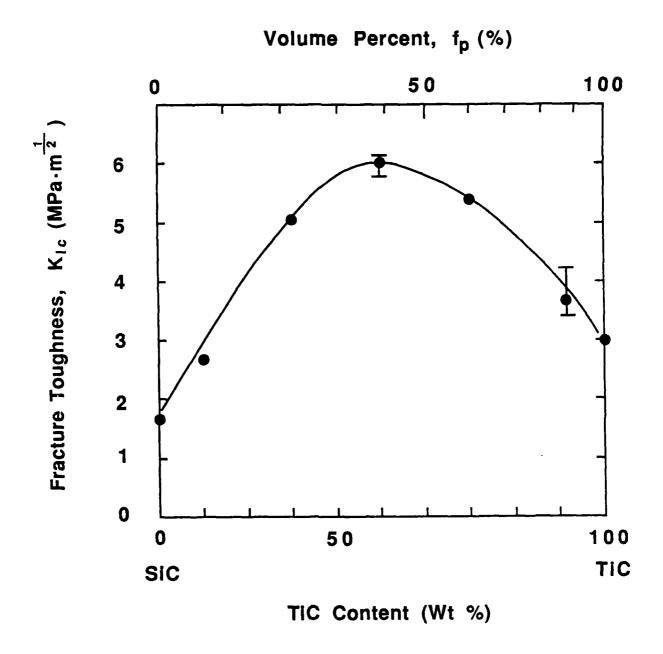


Fig. 16 K<sub>IC</sub> versus f<sub>p</sub> relation of TiC-particulate/SiC-matrix composite.<sup>33</sup>

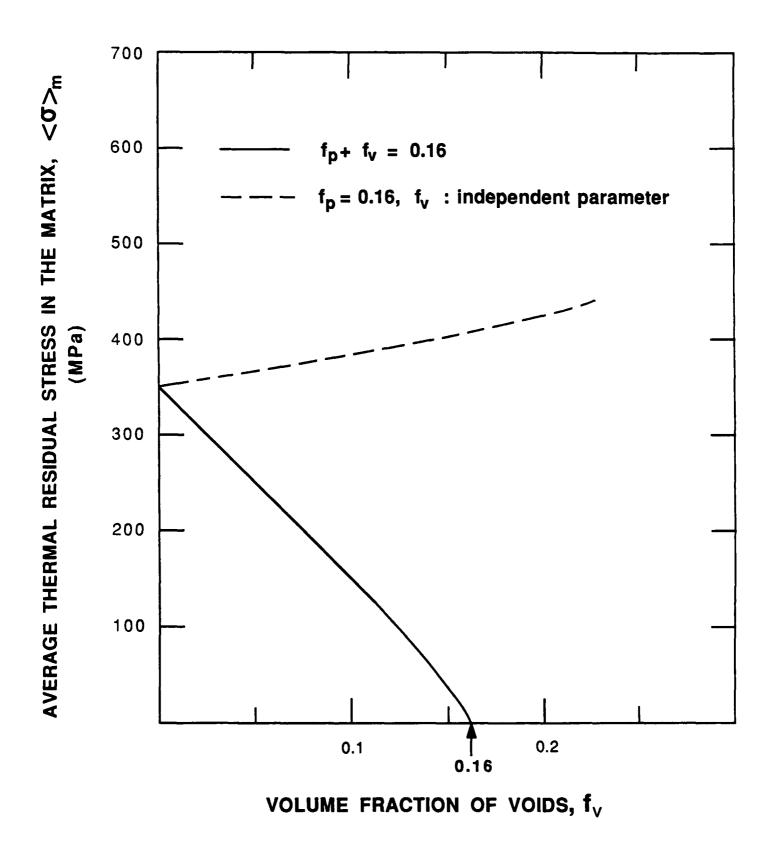


Fig. 17  $<\sigma>_m$  as a function of volume fraction of voids,  $i_v$ . Predicted by eqs. (5) and (6).

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The toughening mechanism of particulate-reinforced/ceramic-matrix composite was attributed to the thermal residual stress field induced by the differential thermal expansions of the matrix and the particulate when the composite is cooled from the processing to room temperature. The measured increase of 77 percent in toughness in a TiB2-particulate/SiC-matrix ceramic composite compared well with the predicted increase of 52 percent.				